Chapter 3

INVESTIGATIVE TOOLS: EXPERIMENTAL METHODS AND PROBES

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3.1 VISION

The 1993 NSF panel report *Atomic Imaging and Manipulation (AIM) for Advanced Materials* (NSF 93-73) concluded that (a) important new science would become accessible as a result of the development of atomic-resolution microscopy, (b) a substantial program in electron microscopy and scanning tip techniques would strengthen U.S. competitiveness, and (c) many user-friendly, low-cost, fast-turnaround compact microscopes were important for rapid progress in much of materials science (Cohen 1993). These conclusions remain valid, but the range of instruments and measurable properties has been extended. Continued development of new tools is critical to the pace of further progress in nanoscience and technology—they provide the "eyes" to see and the "fingers" to manipulate nanostructures. In the nearer term, the greater need is to provide laboratory researchers with the instruments and tools to discover and investigate new chemical, physical, and biological phenomena and applications. In the longer term, those tools will evolve into inexpensive, easy-to-use sensors and/or diagnostic devices with broad applications.

3.2 CURRENT SCIENTIFIC AND TECHNOLOGICAL ADVANCEMENTS

The recent rapid advances in nanotechnology are due in large part to our newly acquired ability to measure and manipulate individual structures on the nanoscale. Whether it be scanning probes, optical tweezers, high-resolution electron microscopes, or other new tools, instruments available to research workers in science and technology now permit them to create new structures, measure new phenomena, and explore new applications. There are limitations for various properties, such as the chemical composition of a single nanostructure and local electronic and thermal characteristics.

Focused Beams

- *Electrons*. Electron microscopy, long the workhorse of science on the sub-micron length scale, is now capable of imaging individual atoms in nanostructures with sub-angstrom resolution (Cowley and Liu 1993). Elemental information is available from electron energy-loss, Auger and X-ray emission measurements with near atomic resolution (Edgerton 1996). New electron based methods have been used to make significant advances in our understanding of magnetic nanostructures (Mankos et al. 1996).

- *Ions*. Ion beams are available with 10 nm resolution and offer some limited analytical capability (Kalbitzer et al. 1993).

- *Photons*. Visible photons are limited by diffraction to spot sizes much larger than a nanometer, unless one operates in the near field (see Scanning Probe section below). X-ray beams might be focused into nanometer spots. Present technology is closer to 1 micron. The limitations are optical elements effective at the X-ray wavelengths and adequate photon fluxes. Rotating anode X-ray sources can provide bright line radiation, but synchrotron radiation is necessary for variable frequency photons. Focusing can be enhanced through capillary X-ray waveguiding (Yamamoto 1997) or, potentially, by the development of nanostructured optical elements.

Electron Microscopy

Rather than focusing an incident beam, electron optics can be utilized to form high-resolution images with the electrons emitted from a surface (Bauer 1990). Image resolution of 12 nm has been reported for photoemission (PEEM) (Ade et al. 1999). Used in conjunction with the new synchrotron X-ray sources, this allows the imaging of nanoscale features with elemental specificity. A variant, which has important applications in the study of magnetic nanostructures, is X-ray magnetic circular dichroism (XMCD) (Stöhr et al. 1993).

Spectroscopic Scanning Probe Microscopes

The inventions of the scanning tunneling microscope (STM) (Binnig et al. 1982) and the atomic force microscope (AFM) (Binnig et al. 1986) have spawned development of a variety of new scanning probe microscopes (SPMs) (Wickramasinghe 1989; Wiesendanger 1994). As a class, the SPMs measure local properties with nanometer-scale spatial resolution by bringing a sharp tip in proximity (1-10 Å) to a solid surface. The proximity of tip and surface enables the SPMs to operate in ambients forbidden to traditional vacuum-based surface analytical techniques. The STM and the AFM were initially limited to monitoring fine scale topography. But the broader class of scanning probes, derived from these initial instruments, allows one to go beyond topography and examine many other local properties, including the following:

- *Electronic structure* by scanning tunneling spectroscopy (STS) (Stroscio and Kaiser 1993), particularly at low temperatures (Bürgi et al 1998; Yazdani et al. 1997).
- *Optical properties* by near-field scanning optical microscopes (NSOM) (Betzig et al. 1991). The NSOM beats the diffraction limit and allows optical access to sub-wavelength scales (50-100 nm) for elastic and inelastic optical scattering measurements (see Figure 3.1), as well as for optical lithography.
- *Temperature* by scanning thermal microscope (SThM) (Majumdar et al. 1993). The SThM uses a temperature-sensing tip (Figure 3.2) to map temperature fields of electronic/optoelectronic nanodevices (Figure 3.3) and to measure thermophysical properties of nanostructures.
- *Dielectric constants* by scanning capacitance microscopes (SCM) (Williams et al. 1989). Since the capacitance of a semiconductor depends on carrier concentration,

the SCM enables the researcher to map out dopant profiles in semiconductor devices with nanometer-scale spatial resolution.

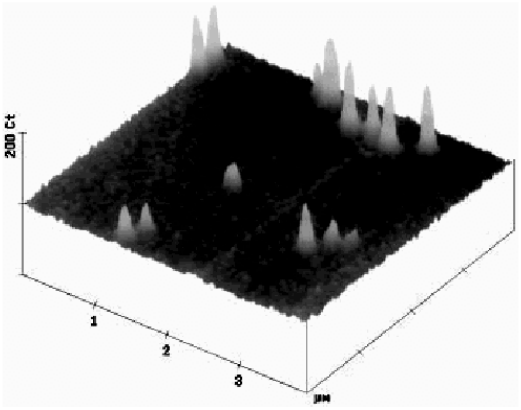

Figure 3.1. Room temperature near-field fluorescence image (4 microns x 4 microns) of single sulfohodamine 101 molecules adsorbed on a silicate glass surface. Each peak is full width half maximum (FWHM) of 100 nm and corresponds to the signal from a single molecule (Bian et al. 1995).

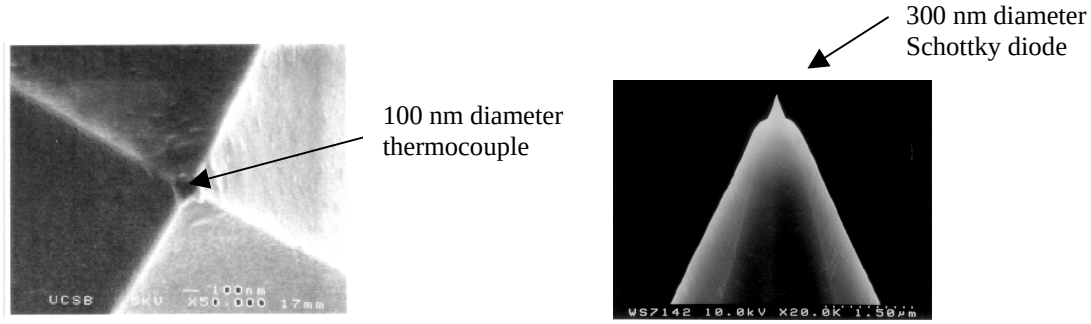

Figure 3.2. Nanofabricated thermocouple (L., reprinted by permission from Luo et al. 1997a, ©1997 American Vacuum Society) and Schottky diode sensors on probe tips (R., Leinhos et al. 1998).

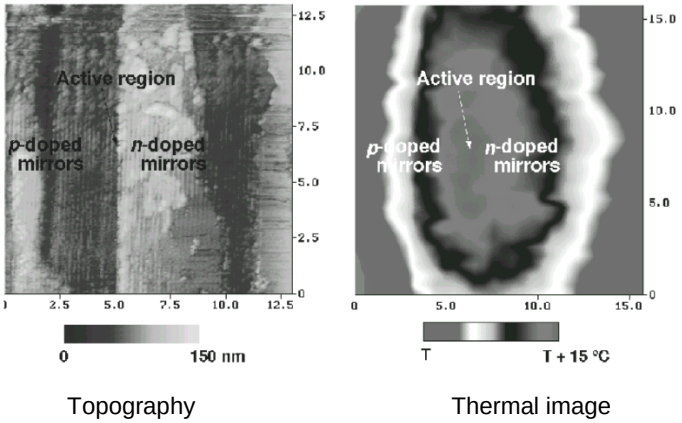

Figure 3.3. Topographical and thermal image of the cross-section of an active vertical cavity laser (reprinted by permission from Luo et al. 1997b, ©1997 American Institute of Physics).

- *Magnetism* by magnetic force and resonance microscopes (MFM) (Hobbs et al. 1989; Rugar et al. 1992). The MFM can image magnetic domains and is already an

integral part of characterizing magnetic storage media. The magnetic resonance microscope can detect nuclear and electron spin resonance with submicron spatial resolution and potentially provides a basis for chemical analysis.

- *Charge transfer and the Helmholtz layer* by scanning electrochemical microscope (SECM) (Bard et al. 1991).
- *Biological molecule folding/recognition* by nanomechanics (Gaub et al. 1997; Colton et al. 1994). Single molecule nanomechanics measurements can provide insights into the molecular phenomena that dominate biological systems and have previously been probed only by measurement of ensemble averages.
- *Chemical information* (Ho et al. 1999; Gimzewski and Joachim 1999; Noy et al. 1997; Knoll and Keilmann 1999).

By providing access to and enabling observation of physical, chemical, and biological phenomena at nanometer scales, SPMs have changed the landscape of experimental research in nanoscience and technology.

Manipulation of Two- and Three-dimensional Nanostructures

Items as small as single atoms and molecules can be manipulated and even exploited as atomic switches (Eigler et al. 1991; Wada 1997). It is interesting to note that atomic manipulation is the smallest possible scale for materials manipulation; we are at a fundamental limit for improving materials behavior through controlling composition and/or structure. There have been many important advances at nanoscale manipulation:

- *Computer-controlled SPM* enables real-time, hands-on human interaction of nanostructure manipulation. In one example, a nanoManipulator (nM) system (Taylor et al. 1993) provides a virtual-environment interface to SPMs; it gives the scientist virtual telepresence on the surface, scaled by a factor of about a million to one. The introduction of direct human-SPM interaction creates not only enhanced measurement capability (for instance, special transducers can provide a sense of touch to the nanomanipulator), but also an automated technology presaging nanofabrication and/or repair of nanostructures. As a demonstration of the educational potential, students in a high school advanced placement biology course have used the nanomanipulator across the Internet to see, feel, and modify Adeno virus particles.
- *Optical tweezers* (Sato and Inaba 1996; Mehta et al. 1999; Kellermayer et al. 1997) provide another new approach to gripping and moving nanometer structures about in three dimensions. This capability has been especially useful investigating particle/ molecular dynamics. A general goal in molecular biophysics is to characterize mechanistically the behavior of single molecules. Whereas past experiments required model-dependent inferences from ensemble measurements, optical tweezers allow a direct observation of the parameters that are relevant to answering the questions, how does a polymer move, generate force, respond to applied force, and unfold?
- *Nanomanipulators* have been reported for use in scanning electron microscopes (SEM) and transmission electron microscopes (TEM). Schmid et al. (1995) have incorporated a manipulating tip, which has 3 degrees of freedom and is controlled to high precision by piezo elements, into a low-energy electron point source microscope.

A piezo-driven TEM specimen holder has been made for observing (with atomic resolution) the mechanical interaction in nanometer-sized crystallites, and the mechanical loading and bending of carbon nanotubes (Kizuka et al. 1998; Poncharal et al. 1999). Using this type of specimen holder, the quantized conductance through individual rows of suspended gold atoms has been observed (Ohnishi et al. 1998). A consequence of combining such levels of manipulation with TEM imaging is that the authors were sure (because they directly imaged the Au atom bridges in TEM) of the number of Au atoms in the particular bridge for which they determined the conductance. Still newer, high performance "nano-manipulators" for SEM and TEM (Yu et al. 1999) have recently been built (see Figure 3.4 and Section 3.7.2 below).

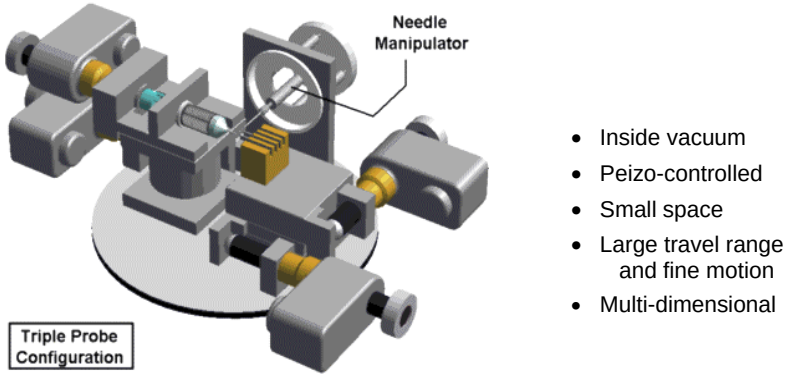

Figure 3.4. Nanomanipulator inside SEM, co-developed by Zyvex and the Rod Ruoff group (see also Yu et al. 1999, reproduced by permission).

Parallel Probe Arrays

Although SPM has been used widely for topographical imaging, atomic/molecular manipulation, and nanoscale lithography, a major drawback is its low raster speed, limited by present cantilever and system dynamics to about 50 Hz/line. To alleviate this problem, several groups are developing arrays of cantilever probes (Figure 3.5) that are individually actuated and controlled (Miller et al. 1997; Minne et al. 1998; Despont et al. 1999). By paralleling the process, they can achieve high-speed nanometer-scale imaging, as well as sub-0.1 μm lithography, on large-scale (1 cm) objects.

In addition to their promise in characterization and fabrication, microfabricated cantilever arrays also show commercial promise as highly sensitive detectors of chemical species (Baselt et al. 1996; Lang et al. 1998).

In-Situ Monitoring and Process Control

Advances in materials processing and fabrication techniques have made it possible to produce superlattice device structures with characteristic layer thicknesses down to several atomic layers and layer interfaces of near atomic precision. Continued demands for improved device performance with simultaneous reduction in production costs have made reproducibility and reliability of superlattice growth vital imperatives. Interfaces must be controlled to atomic dimensions. In situ sensing and feedback control of growth

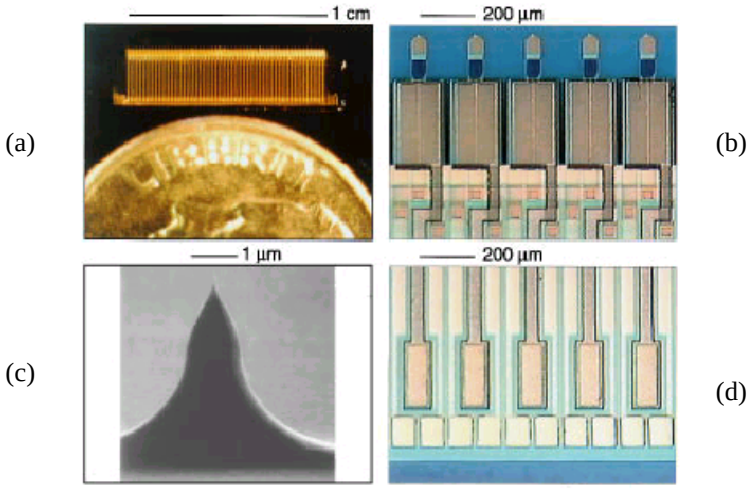

Figure 3.5. An array of cantilevers with integrated actuators and sensors with improved shielding between the actuator and sensor: (a) shows an entire array of 50 cantilevers spanning 1 cm next to a dime; (b) shows a detail of five cantilevers, spaced by 200 µm; (c) is an SEM of a typical integrated single-crystal silicon tip (radius of curvature is below 10 nm); and (d) shows the corresponding electrical contact structure for the cantilevers. There are three leads per device: piezoresistor, ZnO, and tip bias (reprinted by permission from Minne et al. 1998, ©1998 American Institute of Physics).

processes like molecular beam epitaxy (MBE) are essential to reduce the incidence of "nanostructural defects" between adjacent layers. Examples of successful in-situ approaches for monitoring deposition of extremely thin layers include reflection high energy electron diffraction, reflectance spectroscopy, in-situ cathodoluminescence, optical flux monitoring, spectroscopic ellipsometry, photo emission oscillations, absorption band edge spectroscopy, desorption mass spectrometry, vacuum ultraviolet photoionization time-of-flight mass spectrometry, pyrometric interferometry, ultraviolet laser-induced atomic fluorescence, and nonlinear (second harmonic) optical spectroscopies (Schroder 1998).

Nanostructured Materials Characterization

The measurement of nanostructured materials properties is complicated by the presence of aggregated nanostructures. Individual nanoparticles are inherently small, and their compositions and structures are affected by the large number of surface atoms. The particles can be collected into varying degrees of compaction with length scales reaching microns and above (Birringer 1994). Porosity, surface areas, and grain boundaries are susceptible to phase segregation and impurities (Tomkiewicz 1996). Specialized techniques are positron annihilation spectroscopy, small angle neutron scattering, small angle X-ray scattering, wide angle X-ray scattering, extended X-ray absorption fine structure (EXAFS), high resolution electron microscopy, and scanning probes (Edelstein and Cammarata 1996).

3.3 GOALS FOR THE NEXT 5-10 YEARS: BARRIERS AND SOLUTIONS

In general, the goal is development of low-cost, high-resolution, standardized, efficient tools and instruments for manipulation and analysis of nanostructures on surfaces (in two

dimensions) and in three dimensions. The following topics are goals for the next 5-10 years:

- *Instruments for analysis of supramolecules, biomolecules, and polymers*. Miniaturized instruments for the analysis of individual molecule properties will be an area of intense research that will have major impact on health, environment, and national security. Microfabricated chips for DNA analysis (Lemieux, et al. 1998; Kurian et al. 1999) and polymerase chain reactions (Kopp et al. 1998) have already been demonstrated. These are the initial steps towards a full-fledged technology of biomedical microdevices, which will not only study and analyze nucleic acids but also other biological molecules such as proteins and carbohydrates. Chip-based sensing for rapid detection of biological pathogens is a critical area with applications in the food handling/processing industry, biological/chemical warfare, and in early warning for exposure to air- and water-borne bacteria, viruses, and other antigens. In one fledgling example, GMR memory elements are being explored for use as biological array detectors (Baselt et al. 1998). Devices such as these require the integration of biology, biochemistry, and surface science with engineering. It is envisioned that biomedical microdevices will be sufficiently inexpensive to make them readily accessible to a large segment of the population, and commonplace in daily life.
- *3-D structure determination*. Present SPMs are limited to analyzing surface or near-surface properties of solids with nanometer-scale spatial resolution. With the exception of the limited capability in ballistic electron emission microscopy (BEEM) (Bell and Kaiser 1996), sub-surface imaging and truly three-dimensional microscopy with nanometer-scale spatial resolution are not currently available; they are, however, extremely important for future development in nanotechnology. For example, most biological nanostructures are three-dimensional and currently imaged by X-ray crystallography, which is expensive and time-consuming. Even in micro/nanoelectronics, which is progressing towards multilayer three-dimensional structures, 3-D imaging would be very useful. Possible approaches for subsurface imaging include ultrasonic echo imaging, non-linear (multiphoton) optical microscopy, and thermal spectroscopic imaging. It is unclear at present which, if any, technique would be suitable. This area clearly needs emphasis.
- *Nanostructure chemical identification*. Chemical identification of an unknown material is crucial to understanding and predicting its properties. Urgently needed are analogs or alternatives to traditional analytical chemistry techniques—elemental analysis (atomic emission spectroscopy, Auger, XPS); mass spectrometry (MS); vibrational (IR, Raman, HREELS); electronic (UV/VIS, UPS); magnetic resonance (NMR, NQR, EPR)—that will work routinely on individual nanometer-sized structures.
- *Functional parallel probe arrays*. Fabrication of probes designed to measure one property has been amply demonstrated; however, full characterization of a nanostructure requires measurement of many properties. One future goal is achievement of multifunctional probes that provide a "laboratory on a tip," or "nanoscale total analysis." Again, this will require integration of knowledge from engineering, chemistry, physics, and biology. As discussed earlier, it is possible to increase the speed of SPMs by making parallel, individually actuated and controlled

probe arrays. Integration of both multifunction and array technologies is likely necessary for realization of rapid nanoscale diagnostics.

- *Standardization and metrology.*
  - Locating and maintaining a position with nanometer accuracy and precision are still difficult, especially if coupled with the requirement to achieve those goals across samples of centimeter dimensions. This is one of the crucial issues that must be solved if commercial nanoelectronic device fabrication is to be realized.
  - Uniform-size nanoparticles of known size and composition are needed for the standardization and calibration of nanoscale measuring instruments.
  - The importance of making measurements on a common set of calibration particles in order to develop reliable standards was underscored by specialists in particles in gases, particles in liquids, particles on surfaces, and mass spectroscopy who came together during a recent DOE Workshop on Instrumentation for Nanoparticles (U. of Minnesota, Dec. 1998). Goals in the next 5-10 years include development of particle-size calibration standards of 3 nm, 10 nm, and 30 nm sizes; improvements in measurement methods for nanometer-size particles, including modeling of the instrument, uncertainty assessment, and improved data analysis methodology; and quantification of uncertainty in TEMs, differential mobility analyzers, and small angle X-ray equipment for measurements over these size ranges. A real-time size distribution analyzer for nanoparticles is needed as a process monitor during processing. Additionally, there is a need for a particle classifier to select nanoparticles into narrow size fractions (Chen et al. 1998).
  - There is an opportunity to define fundamental standards based on the creation of atomically controlled and measured structures (see schematic in Figure 3.6). Quantized electron devices may provide known electrical currents. Macromolecules/clusters of known mass (having a countable number of elemental constituents) may provide building blocks of a gram.
- *New Nano-Manipulators.* Nanotechnology has a goal of 3-D manipulation of chemical moieties to build molecules/clusters and then to assemble them into larger devices and materials. Achieving this requires combining techniques of chemical synthesis with engineering methods that wield atomically precise positional control. Manufacturing technologies such as microelectromechanical systems (MEMS) are potentially capable of producing higher degree-of-freedom micromachines that can exert molecular-level positional control and bridge mesoscopic extremes in handling nanoscale and microscale components. Extension of MEMS into nanometer-sized electromechanical structures (NEMS) will achieve that capability. In combination with chemical functionalization schemes and self-assembly concepts, MEMS/NEMS will form an essential generation of hybrid machines for subsequent stages of nanotechnology development.

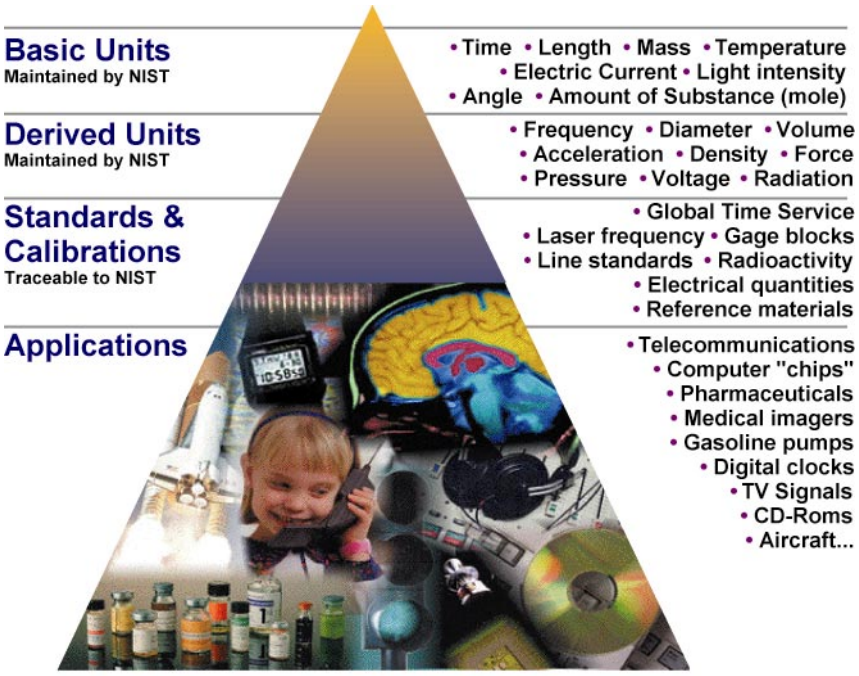

Figure 3.6. Areas for nanotechnology standards (courtesy M. Casassa, NIST).

- Other five-year goals include the following:
  - Batch-fabricated integrated measurement and lithography systems
  - Further investigation into top-down/bottom-up fabrication
  - Non-SPM probes that use electrons, ions, etc. (atomic-scale electron microscopy)
  - Intelligent analysis systems for medical, environmental, and defense applications
  - In-situ, nondestructive monitoring techniques for submonolayer control of superlattice growth

3.4 SCIENTIFIC AND TECHNOLOGICAL INFRASTRUCTURE

Building new infrastructure to support development of new tools and experimental methods must take into account the following considerations:

- The development of new instrumentation for nanostructure measurements, especially the scanning probes, has and will depend critically on synergestic work between university/government researchers (new ideas) and industrial developers (commercial realization). A government investment strategy must encourage and reward multidisciplinary collaborations among these communities.
- The small amount of material in, and the tiny size of, nanostructures frequently requires the use of special, expensive facilities: high-intensity synchrotron radiation sources, thermal neutrons, and high-energy electron beams (lithography and high-resolution electron microscopy). Adequate support for these facilities is important, both to create them and to provide affordable access to visiting researchers.
- While the scanning probes can be sufficiently inexpensive and routine for single-investigator acquisition and usage, state-of-the-art utilization of the probes can

require highly specialized knowledge and apparatus. There should be a reasonable number of scanning probe analytical centers where that kind of knowledge and apparatus are available to visiting researchers. Those centers should also be expected to continue advancement of scanning probe capabilities.

- The need for a database of information on proximal probe instrumentation, recipes for sample and probe preparation, standards and calibration procedures, and image analysis algorithms is becoming critical. An Internet-based information exchange could make this knowledge available to all potential users.
- Instrumentation development is not highly valued in the United States. To achieve the sophisticated instruments of tomorrow, it will be necessary to build the interest, knowledge base, and skill level of today's students. Toward this end, it will be helpful to create scholarships and fellowships to attract high-caliber high school students and post-docs interested in instrumentation and nanofabrication.

3.5 R&D INVESTMENT AND IMPLEMENTATION STRATEGIES

- Significantly increase investment for development of nano-instruments/tools that are low-cost, user-friendly, accurate, and reliable and user facilities that enable nanotechnology development. Early investment in instrumentation will yield benefits in all aspects of nanostructure science and technology.
- Foster industry-university-national laboratory cooperation in developing and commercializing nanoinstruments and tools.
- Ensure adequate support for high-performance beam sources (synchrotron light, neutron, and electron beams) for analytical facilities that provide affordable, state-of-the-art capabilities to the research community.

3.6 PRIORITIES AND CONCLUSIONS

The advancement of nanoscale science and technology can be facilitated by the development of nanoscale measurement instruments with improved capability. A major priority is to extend research instrument capability into low-cost, accurate, and reliable systems that can be used by researchers to explore new phenomena and to characterize fully nanostructured materials. Enabled by this capability, nanoscience advances will rapidly transition to applications in healthcare, food safety, environmental safety, law enforcement, and national security.

3.7 EXAMPLES OF CURRENT ACHIEVEMENTS AND PARADIGM SHIFTS

Several examples of new instruments and their utilization at nanoscale are presented below, including manipulation of single molecules and nanotubes, near-field optical and surface force microscopy, and observed nanoscale images on surfaces.

3.7.1 Single Molecule Manipulation and Measurement

Contact person: James Murday, Naval Research Laboratory

Tools to manipulate and measure single-molecule properties provide critical capabilities:

- Biology, medicine and healthcare will be revolutionized by the ability to manipulate the chemical/physical basis of living systems originating in the behavior of molecules at nanometer scales (see Chapter 10).
- As miniaturization continues, electronic structures will reach molecular sizes; carefully positioned single molecules can provide needed properties (see Chapter 8).
- Structural polymers, adsorbents, and supramolecular catalysts (e.g., enzymes) depend on molecular folding, shape, and reconfiguration (see Chapter 7) and can be designed for greater efficacy.

In the past, measurements of molecular behavior were necessarily ensemble averages; it was not possible to probe an individual molecule. While averaging techniques are very powerful, they mask detailed information necessary to fully understand the properties of matter, and more importantly, to enable full exploitation of the molecular behavior. The revolutionary advances in instrumentation featured in this chapter are providing exciting entrees into the single molecule world. Examples follow:

- *Carbon nanotubes.* Early theory predicted outstanding electrical and mechanical carbon nanotube properties that are now confirmed by measurements of individual nanotubes (Figure 3.7).

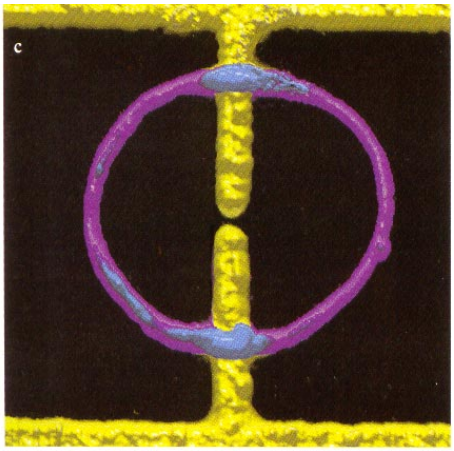

Figure 3.7. Carbon nanotubes circling electrical contacts (reprinted by permission from Dekker 1999, ©1999 American Institute of Physics).

- *Molecular recognition.* Much of biochemistry, including the immunoresponse critical to health, depends on molecules recognizing and binding to specific sites. Direct force-displacement measurements on bound molecules are now possible (Figure 3.8). This has led to a revolutionary approach to molecular detection—the force discrimination assay—where the recognition force between two biomolecules (antibody/antigen or complementary DNA strands) provides highly selective and sensitive detection.

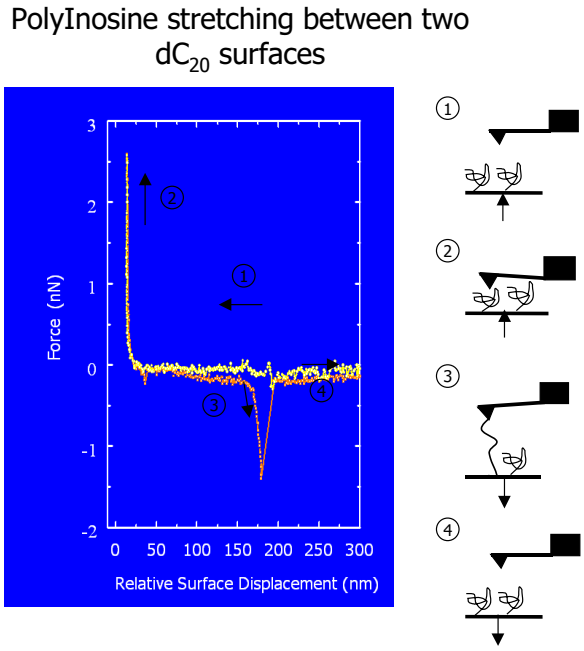

Figure 3.8. Force microscope measurement of complementary DNA binding (reprinted with permission from Baselt et al. 1996, ©1996 American Vacuum Society).

- *Molecular motors.* Molecular motors are responsible for DNA transcription, cellular transport, and muscle contraction. The new microfabricated tools enable us to isolate, understand, and exploit these motors as new actuators for nanoelectromechanical tools—much smaller versions of microfabricated tools. This may lead to artificial biological devices, embedded in the body and powered by the same ATP that fuels normal body processes (Figure 3.9).

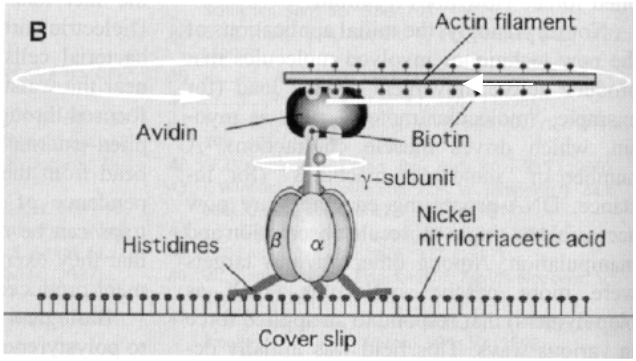

Figure 3.9. F_1-ATPase with actin filament mounted on a glass substrate (reprinted by permission from *Nature*, Noji et al. 1997, ©1997 Macmillan Magazines Ltd.).

- *Molecular folding*. A fundamental research problem in biochemistry is protein folding: how does a protein "know" its final configuration and achieve it quickly? Folding of structural polymers (e.g., crystallization, lamella formation) presents similar quandaries. A plethora of new techniques are providing direct molecular measurements of folding forces and dynamics, including optical tweezers and others (Figure 3.10).

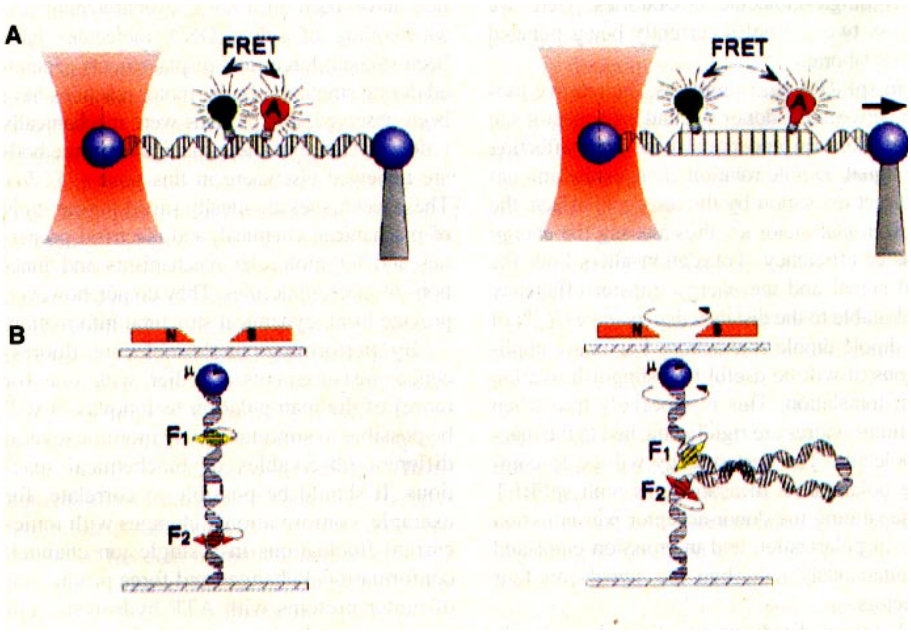

Figure 3.10. Optical tweezer and magnetic bead manipulation coupled with fluorescent probes (FRET) (reprinted with permission from Weiss 1999, ©1999 American Association for the Advancement of Science).

3.7.2 Nanomanipulator Inside a Scanning Electron Microscope

Contact Person: Rod Ruoff, Washington University, St. Louis

Innovations in manipulation and measurement of nanostructures are largely based in university and government laboratories; industry pays close attention to their discoveries and commercializes those that are most promising. As an example, a university-industry interaction between Washington University in St. Louis and Zyvex, a small business, has led to a new tool for manipulating nanoscale objects while simultaneously imaging with a SEM (illustrated in Figure 3.4 above). With this device, pulling, bending, and buckling of nanotubes into the third dimension are possible. The manipulator features a wide translation range, reasonable precision, small size, low-cost, and rapid assembly. Coarse 3-axis linear motions up to 6 mm and single-axis 360 degree rotational probe motion are provided by vacuum-prepared stainless steel stages driven by similarly prepped piezo actuators. An integral X-Y stage guides motion parallel to the plane of the SEM stage, and a separate Z-axis stage is used for motion along the SEM beam axis. Rotational motion normal to the beam is accomplished using a picomotor rotating actuator mounted atop the Z-stage. A four-quadrant piezo tube serves both as a support for the rotating tip and as a fine motion actuator in order to provide continuous motions augmenting the picomotor stepper action. Angular step sizes of < 0.02 degrees with a maximum rotation rate of ~20 degrees/s, and spatial resolution of the piezotube of better than 0.1 nm are achieved.

Figure 3.11 shows SEM images of a single multiwalled carbon nanotube being stress-loaded after it has been attached across two atomic force microscope cantilevers; the imaging enables and confirms the attachment of a single tube. It also provides direct visual observation of tube dynamics.

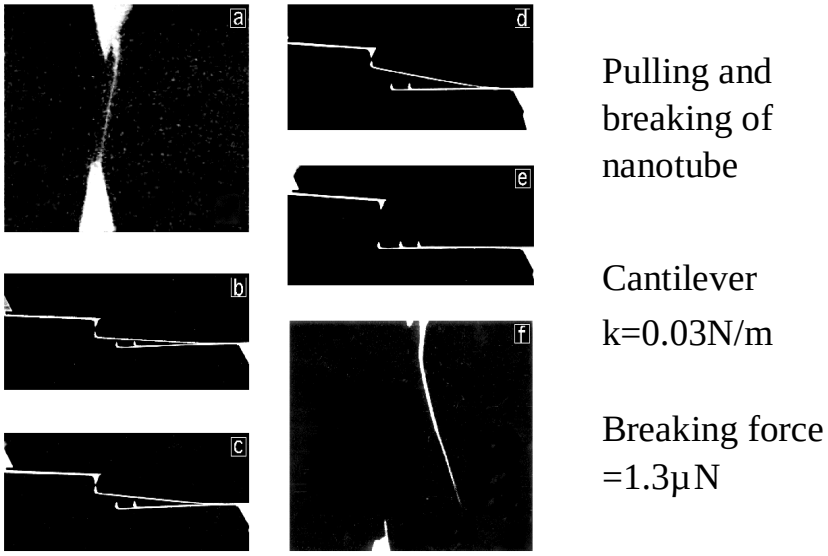

Figure 3.11. SEM images of a single multiwalled carbon nanotube being stress loaded and breaking away after it has been attached across two atomic force microscope cantilevers (Yu et al. 1999, reproduced by permission).

3.7.3 Multifunctional, Combined Near-Field and Surface Force Microscopes

Contact persons: Daniel van der Weide, University of Delaware, and James Murday, Naval Research Laboratory

Optical microscopy has been an essential tool in the scientific arsenal for centuries. Since the middle of the 1800s, the diffraction limit has constrained the resolution of optical images to the wavelength of light—about 0.5 micron in the visible spectrum. Development of scanning tunneling microscopy and atomic force microscopy in the 1980s provided imaging with three orders of magnitude better resolution. However, the basic physics in every form of microscopy limits what it measures. STM is predicated on electron tunneling; its images are defined by tunneling physics or by relaxation processes associated with the injected low-energy electrons. AFM has a broader range of capabilities; it can respond to a wide range of forces between tip and substrate—magnetic, Coulombic, dispersive, friction, core repulsion, etc. Optical imaging would complement STM/AFM images. Diffraction is a far field radiation effect; near field microscopy avoids the diffraction limit by working close to the sample. Several variants of near field scanning optical microscopy (NSOM) have been developed that utilize small apertures and/or tip antennae. Demonstrated visible light image resolution is ~10 nm.

Near field microscopes are not limited to visible wavelengths. A recent innovation has been the combination of near field and force microscopes (Figure 3.12). A miniaturized coaxial cable is fabricated onto a force microscope cantilever, terminating at a tip with nanometer dimensions. This geometry produces tiny probes with no cutoff frequency, is shielded to limit Coulomb interactions, and simultaneously probes topography (via force) and time varying electric fields (up to several GHz via near field).

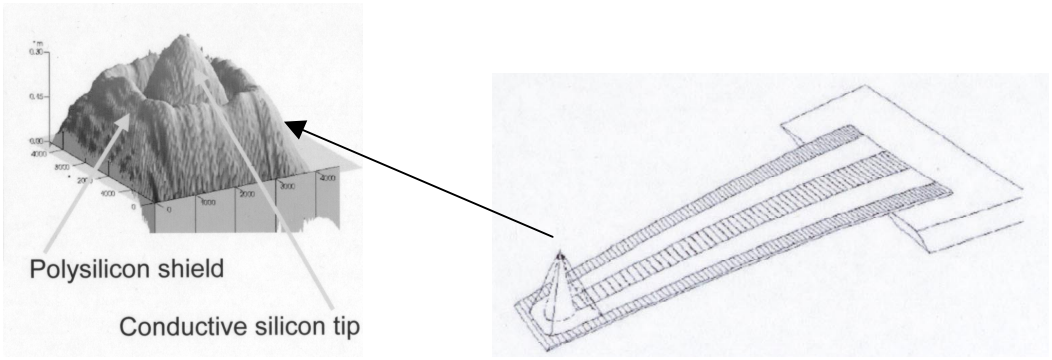

Figure 3.12. Near field antenna probe (after van der Weide and Neuzil 1996, reprinted by permission, ©1996 American Vacuum Society).

As an illustration, the image of a non-linear transmission line with ~100 nm topology is shown in Figure 3.13, along with the 30 ps waveforms detected at the specified point. This tiny near field antenna probe can operate in several modes: detection, excitation, reflection and transmission. It is a powerful new approach to the study of items as diverse as millimeter-wave electronic circuits and nerve cells.

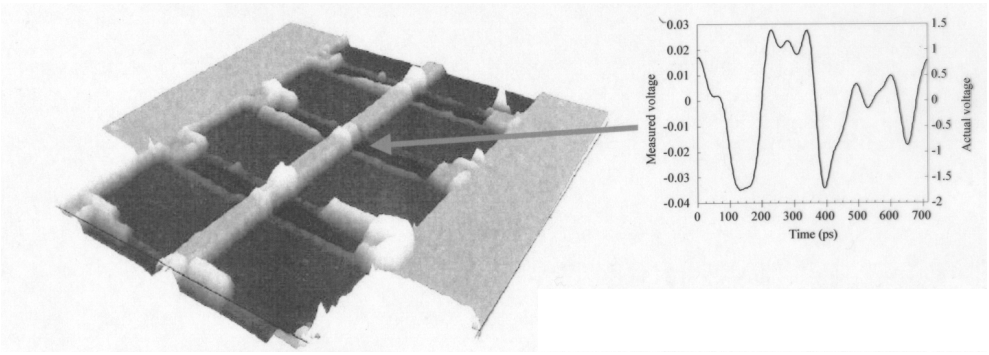

Figure 3.13. Near field antenna correlated measurement of topography and waveform (reprinted by permission from van der Weide 1997, ©1997 American Institute of Physics).

3.7.4 Image of Nanostructures on Surfaces

Contact person: P. West, ThermoMicroscopes

Figure 3.14 shows a monolayer of red blood cells on a mica substrate.

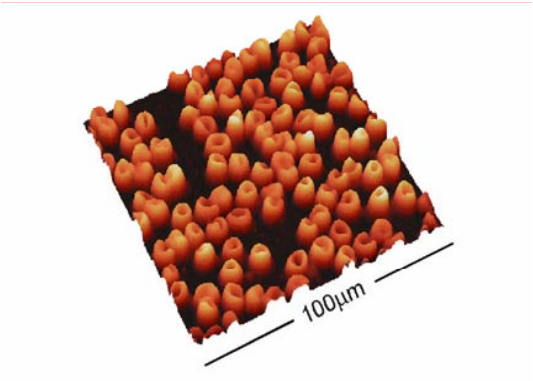

Figure 3.14. STM image and measurement of red blood cells (courtesy L. McDonnell).

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